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COMPLETE SPECIFICATION

Improvements relating to Filled Polyolefine

We, W. R. GRACE & Co., a Corporation organised and existing under the laws of the State of Connecticut, United States of America, of 7 Hanover Square, New York 5, New York, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to filled polyolefine compositions and their production and use.

The use of inorganic fillers as extenders or reinforcing agents for rubbers and some synthetic resins is well established. Most attempts to use fillers in a similar manner to extend or reinforce the more crystalline polyolefins, however, have met with failure, brittle products being generally obtained, even with moderate filler concentrations. Previous attempts have been made to produce polyethylene/filler blends using conventional inert fillers alone, and although occasionally these blends are found to have greater tensile strength than the unfilled polymer, most of them are too brittle to be useful as general purpose resin-base compositions. On the whole, reported studies of polyethylene/filler blends indicate that satisfactory products are rarely obtained. Some inorganic materials can be blended into polyethylene as pigments, but in amounts too small to serve other purposes.

Fillers are now widely used as extenders for glass fibre reinforced resins. In most cases of this type, the filler does not impart any reinforcement, but does have a favourable effect on cost, shrinkage, thermal effects, surface properties, and flow properties. However, apparently no other material is reinforced by fillers to the same extent as the natural or synthetic rubbers.

It has now been found that polyolefines, in particular polyethylene of very high molecular weight can tolerate high filler loadings without becoming brittle, unlike conventional polyethylene having a molecular weight of around 60,000 to 100,000, which yields brittle products at relatively low filler concentrations. Moreover plasticisers can also be incorporated to provide good flow characteristics and to facilitate mixing, without causing excessive loss of strength. Accordingly, the compositions of the invention comprise 5 to 90% of a polyolefin, particularly a polyethylene, of molecular weight sufficiently high to give it a standard load melt index (as defined below) of substantially zero, 5 to 90% of inert filler material, and 5 to 90% of plasticiser (as hereinafter defined), all percentages being by volume.

The high molecular weight polyolefine will usually be polyethylene, especially of the high density type (0.93 to 0.97) and the invention will be described in more detail with particular reference to such polyethylene. Preferably the polyethylene has a density of 0.93 to 0.97, a high load melt index of at most 1.8, especially 0.01 to 1.8, and a viscosity of at least 4.0, especially at 9.3 to 4.0, measured on a solution of 0.02 gram of polymer in 100 grams of decalin at 130°C. However other polyolefines of high molecular weight can be used, including low density polyethylene, polypropylene, and copolymers of ethylene and butylene.

Each of the above components is essential for the attainment of desirable physical properties. The high molecular weight polymer confers strength and flexibility on the composition. Compositions containing high concentrations of the high molecular weight polyethylene generally have the best properties, but the polymer of high molecular weight can be modified by blending with conventional polyethylene with little sacrifice of quality of physical properties. Plasticisers enhance elongation and flexibility, but their primary

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role is to raise the melt index and thus produce a processable composition and facilitate the initial dispersal of filler into the matrix. Although blends of polyethylene and plasticiser are generally incompatible and under ordinary conditions the plasticiser exudes from the sample, the inorganic fillers are found to prevent this exudation. Generally speaking it has been found that the greater the surface area of the filler, the greater amount of plasticiser that can be incorporated. These fillers also are very low in cost and thus serve as cheap "extenders" for the more costly organic components.

A wide variety of inexpensive, finely divided materials is available for use as fillers. The following types are included purely as examples: (a) carbon blacks, (b) metal oxides and hydroxides, especially aluminium oxide and hydroxide, and also silica and hydrated silicas, (c) metal carbonates, and (d) metal silicates and aluminates; naturally occurring clays and mica; precipitated silicates and synthetic zeolites.

Preferred fillers are kaolin, calcium silicate, calcium carbonate, magnesium carbonate, magnesium oxide, stannic oxide, mica, glass beads, glass flake, asbestos, carbon black, silica, aluminium polysilicate, montmorillonite, attapulgit, talc and wood flock.

Any of the above inorganic products can be modified to produce an organophilic material. It has been found that fillers of very high surface area are very effective in retaining the plasticiser, but generally give products with unmeasurably low melt indices. Such fillers can however be used in combination with fillers of low surface area to help to retain the plasticiser.

Various inorganic materials generally considered to be soluble in water are also suitable fillers for use in this invention. The use of such fillers is especially valuable where a porous product is desired, the filler being easily extracted from the polyethylene-filler plasticizer product with water, which greatly reduces the cost of fabrication. Examples of water-soluble materials suitable for use as fillers include the following: (a) inorganic salts, e.g. sodium, potassium and calcium chlorides, (b) acetates, e.g. sodium, potassium, calcium, copper and barium acetates, (c) sulphates, e.g. sodium and potassium sulphates, (d) phosphates, e.g. sodium phosphate (Na_3PO_4), and potassium phosphate, (e) nitrates, e.g. sodium and potassium nitrates, and (f) sugar.

By the term "plasticizer" as used herein is meant a material which will perform the following functions. Firstly, the addition of the plasticizer will improve the processibility of the composition, i.e. lower the melt viscosity or reduce the power input required to compound and to fabricate the composition. As explained more fully hereinafter, the melt index is an indication of the processibility of the composition, the melt index increasing as the molecular weight and viscosity decrease. Similarly, a torque decrease indicates a lower melt viscosity and greater ease of compounding (faster mixing cycle, lower power requirements, and better dispersion of filler). Secondly, the plasticizer will improve the flexibility of the final composition. The improved flexibility is reflected in such measurements as the elongation at failure, the elongation at yield point, Spencer impact, and tension impact. A third and optional function of the plasticizer is its use in the production of porous objects. The plasticizer is the component of the polyethylene/filler/plasticizer composition that is easiest to extract. The extraction can be performed with any of a large number of commercially available organic solvents, the particular solvent used depending upon the plasticizer. It is especially advantageous, however, to use a plasticizer which is soluble in water. By using a water-soluble plasticizer, the extraction process will be more economical owing to the low cost of water in comparison to that of organic solvents. The extraction process will also be much safer as there will be no fire or toxicity hazards encountered. In addition, the water-soluble plasticizer will give to the polyethylene or the polyethylene filler substrate a hydrophilic (water wettable) character which is especially desirable in situations where the intended use of the porous product requires that the product meet minimum conductivity and permeability standards.

It should be noted that the plasticizer used in this invention does not necessarily have to dissolve in the polymer. This is in contrast to the function of a plasticizer as generally understood. However, any liquid of low volatility meeting the above three requirements is suitable for use in this invention.

Examples of the numerous suitable plasticizers includes:

a) Esters, e.g. sebacates, e.g. dibutyl and dioctyl sebacate, fumarates, e.g. dioctyl fumarate, phthalates, e.g. diisodecyl phthalate, stearates, e.g. butyl stearate; epoxy compounds, e.g. octyl epoxy tallate; polyesters, e.g. polyester glycol.

(b) Phosphate esters.

(c) Hydrocarbons, e.g. paraffin oil, paraffin wax and low polymers such as polyisobutylene and polybutadiene.

(d) Chlorinated hydrocarbons, e.g. chlorinated biphenyl.

(e) Sulphonamide, coumarone-indene and asphalt.

(f) Polymeric materials, such as ethylene/vinyl acetate copolymers.

Examples of the numerous suitable water-soluble plasticizers include:

(a) Glycol, glycol ethers and esters.

(b) Glycerine and glycerol monoacetate.

(c) Diethylene glycol, diethylene glycol ethers and esters, and triethylene glycol.

(d) Polyethylene glycols (molecular weight range 400 to 20,000).

(e) Propylene glycol, dipropylene glycol.

(f) Polypropylene glycol (molecular weight range 260 to 1200).

(g) Trimethylene glycol, tetramethylene glycol and 2,3-butylene glycol

(h) Alkyl phosphates, (e.g. triethyl phosphate).

(i) Water-soluble polymeric materials, such as polyvinyl alcohols, partially hydrolysed polyvinyl acetate, polyacrylic acid, and polyvinyl pyrrolidone.

It is also possible to make the polyethylene product of this invention using various combinations of the above-mentioned plasticizers, e.g. a water-soluble plasticizer and a water-insoluble plasticizer may be used with a suitable filler and high density polyethylene. Such combinations are intended to be within the scope of this invention. Most of the work leading to the invention has been carried out using commercial particulate high molecular weight polyethylene, having a standard load (2160 g.) melt index of 0.0, a high load (21600 g.) melt index of 1.8, a density of 0.95, and a viscosity of 4.0 measured on a solution of 0.02 g. of polymer in 100 g. decalin at 130°C. This polymer can be prepared by the method given in U.S. Specification 2,825,721 using an ammonium fluoride-treated chromium oxide catalyst. When the term "particulate" is used herein, it refers to the aforesaid polymer. However, any commercially available polyethylene having a standard load melt index substantially zero can be used satisfactorily. Many of the examples to be described used polyethylene having a standard load melt index of 0.00, a high load melt index of 0.01, and a viscosity of 9.3 on the same basis.

The standard load melt indexes referred to herein are a measure of flow under standard conditions of temperature, pressure, and time through an orifice of defined diameter and length as specified in ASTMD 1238-52T. The rate of extrusion in g/10 minutes is the melt index, and it is used to indicate the average molecular weight of a polymer. The lower the molecular weight of a polymer, the more rapidly it extrudes, and therefore melt index increases as molecular weight decreases. By "high-load melt index" (HLMI) is meant melt index determined by the procedure of ASTM-D-1238-52T, except that a weight of 21,600 g. is used.

As has already beeen decribed, the high molecular weight polyethylene can be blended with standard commercial lower molecular weight polyethylene, bearing in mind that if the overall molecular weight of the blended polymer becomes too low, the product is apt to become brittle. Thus the compositions of the invention may additionally comprise 1 to 80% of polyethylene of standard load melt index 0.01 or higher. A preferred composition of this type comprises 5 to 85% of polyethylene of zero standard load melt index, density 0.93 to 0.97, and a high load melt index of 1.8; 5 to 40% of polyethylene of standard load melt index 0.7; 5 to 85% of inert filler material; and 5 to 85% of plasticizer. Another such composition comprises 5 to 85% of polyethylene of zero standard load melt index, density 0.93 to 0.97, and a high load melt index of 0.01; 5 to 75% of polyethylene of standard load melt index 0.7; 5 to 85% of inert filler material; and 5 to 85% of plasticizer. Extensive studies were carried out using as a blending agent commercial lower molecular weight polyethylenes, for example GREX brand of polyethylene (W. R. Grace & Co.) having a standard load (2160 g.) melt index of 5.0 and a viscosity of 1.5 on measured on a solution of 0.1 g. polymer in 100 g. decalin, and also having a standard load melt index of 5.0 and a viscosity of 1.5 on the same basis.

End products based on the compositions of the invention are comparatively very cheap. The material cost for a typical composition (consisting of 40% by volume high molecular weight polyethylene, 30% by volume kaolin clay and 30% by volume paraffin oil) would be less than 0.06 U.S. dollars for the volume of material equivalent to one pound of high density polyethylene.

One technique employed to produce and test the new composition can be described as follows. In most cases the various components were premixed at room temperature in a V-blender, though in some cases the filler and plasticizer were slurried together at room temperature in a volatile solvent which was evaporated before the materials were combined with the polyethylene. The polyethylene/filler/plasticizer "dry blends" were then mixed in a Brabender Plastograph which continually recorded the torque required for the mixing process. At the same time, the temperature of the mixture was measured by a thermocouple. Thus, changes in the melt viscosity of the mixture were observed and the mixing of characteristics of different blends compared.

The following mixing procedure was also found to be satisfactory. The polymer was added to the mixing chamber which was preheated to 180°C. When the polymer fluxed, the filler was added. In those areas where the initial portions of the filler produced extremely high torques, portions of the plasticizer were added to bring the torque down before the rest of the filler was added. Generally five minutes was allowed to melt the polymer and add the filler before the recorded milling cycle was carried out. It was found that when the filler is added as a dry powder

it frequently accumulates in "dead spots" on the blades or in the mixing chamber. For this reason, a master batch technique was often used. This required no difference in technique. The filler was simply mixed into the molten polymer at a high concentration to form the master batch, which was then diluted with more polyethylene and plasticiser by the same process. Any unblended filler adhering to the master batch blended into the final product without any trouble. The sample and master batch milling cycles can be carried out under a variety of conditions. In the experimental work performed, samples and master batches were mixed at 30 or 90 RPM, in air or nitrogen and for various times.

The final product blends can vary widely in overall composition, depending in part on the desired physical properties of the product.

Careful testing was carried out on all experimental samples. The polyethylene/filler/plasticizer blends were normally pressed in standard fashion common to the art on a hydraulic press into 0.020 inch (nominal thickness) sheets for testing, which were pressed at 176°C. for three minutes at about 1000 p.s.i.

These sheets were used to determine relative flexibility and tear strength by hand, and to determine tensile properties with an Instron tensile tester. Obviously, no absolute scale for flexibility or tear strength can be set with hand tests, but samples which were very poor were quickly eliminated. Normally any sample which broke completely upon being bent 180° or less was rejected.

The samples were also roughly classified according to their resistance to tearing. The best samples resisted tearing under all conditions and showed signs of cold drawing throughout the length of the tear. With some other samples it was difficult to initiate a tear, but once started the tear propagated rapidly. Poorer materials afforded no initial resistance and the least satisfactory samples cracked apart.

The tensile properties were measured by an Instron tensile tester, which continually records stress as it pulls the sample at a constant rate of strain, using straight samples 0.25 inch wide cut from the 0.020 inch sheet and clamped at points 5.1 cm apart and tested at a rate of 1 inch or 50% per minute.

Three samples of each composition were tested. The results were computed to give the 1% modulus (i.e. the ratio of stress to strain at 1% elongation), the stress and elongation at the yield point (S_{yp}, E_{yp}) and the stress and elongation at failure (TS,EF). As a sample was elongated during the test, the stress intially built up rapidly. At the yield point there was a decrease in stress as the sample started to cold draw. The S_{yp} was measured just before this change. The sample was then elongated, primarily by cold drawing, to the point of failure. The reported tensile strength was then calculated on the basis of the stress prior to failure and the original dimensions of the sample. The tensile modulus (TM) values indicate the stiffness of the samples. The S_{yp} is a measure of the strength of the sample and indicates any reinforcement due to the presence of the filler.

A derived function, the "work," is also included. The value given is one half the product of the S_{yp} in p.s.i. and the elongation at yield point in inches. The true value of work done would be given by the area under the stress strain curve up to yield point. The deviation of the true value from the derived value (or the true area from that of a right triangle) will depend on the initial rate at which the stress increases without strain (i.e. the TM). Therefore values of "work" should not be compared without comparing TM as well.

Spencer impact (SI) (the resistance of thin films to rupture) was measured on samples 5 to 8 mils. thick and 5 inches in diameter. Some samples did not fail when they were tested, and hence no value could be reported even though the samples were very good.

The melt index of the samples (ASTM-D-1238T, 1952) was measured at 190°C using weights of 2160 g. ("standard load" melt index, SLMI, or simply MI), 7840 g. ("medium load" melt index, MLMI) and 21,600 g. ("high load" melt index, HLMI).

The thixotropy index, a relationship indicating the change in apparent viscosity at different shear rates, HLMI/MLMI, is also given for some of the samples.

The application of the invention will be made clearer by considering an illustrative system consisting of high molecular weight particulate polyethylene (melt index 1.8 HL), kaolin as filler, and heavy mineral oil (viscosity 335—350 SSU at 100°F.) as the plasticiser as shown in Table I.

TABLE I

High Molecular Weight Particulate Polyethylene Filled with Aluminum Silicate and Plasticized with Paraffin Oil

Composition, % by Volume														
Poly-ethyl-ene	Kaolin (Alum-inum Sili-cate)	Oil	Type of Paraffin Oil (vis-cosity in SSU at 37.8°C	Melt Index SL	ML	HL	Thixo. Index	Tensile Modulus, psi.	Stress at Yield Point psi.	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi	Elongation at Failure %	Spencer Impact psi.
100	0	0	—			1.8	—	150000	3620	10.8	391	3300	693	.261
100	0	0	—		.12	1.3	11	198000	3530	13.5	476	5450	785	*c*
90	0	10	559*a*	.004	.16	1.8	11	115000	2710	21.1	572	3380	875	.305
82.5	0	17.5	335—350		.12	2.3	19	65000	2270	43.4	988	4300	1140	*c*
77	0	23	„		.45	6.6	15	47000	1770	50.2	888	3640	1250	*c*
67	0	33	„		1.1	16	15	38000	1430	42.9	614	2560	1230	.326
80	6	14	559*a*	.013	.19	2.5	13	110000	2370	22.8	540	2660	800	.289
90	10	0	—	.003	.11	1.1	10	318000	3470	7.4	257	3730	730	.276
70	12	18	559*a*	.018	.33	3.4	10	116000	2230	30.0	670	2650	800	.306
80	20	0	—	0	.04	.50	13	390000	3630	3.9	142	3060	5	.270
55	21	24	559*a*	.010	.23	4.3	19	203000	2850	22.0	628	2580	500	.258
50	20	30	335—350		.73	5.5	7.5	87000	1660	25.0	415	1610	850	.253
40	20	40	„		2.9	43	15	66000	1160	19.5	227	1540	1190	.224
50	30	20	„		.10	1.9	19	139000	2180	10.8	236	1710	175	.230
40	30	30	„		.35	8.6	25	119000	1610	12.6	203	1410	54	.172
40	30	30	125—135	.005	.11	4.6	42	126000	1510	13.7	206	1360	54	.062
30	30	40	335—350		1.7	59	35	80000	1100	10.9	120	960	68	.148
30	30	40	„	.003	.20	24	120	87000	840	19.0	160	690	600	.093
30	30	40	„	.002	.16	34	210	55000	885	31.2	276	710	470	.093
30	30	40	335—350	.007	.03	16	530	101000	1010	14.6	146	950	33	.067
40	40	20	„		.002	.12	60	176000	2270	6.9	157	2010	38	.178
30	40	30	„		0	.10	—	139000	1620	5.2	84	1400	68	.126
30	50	20	„		0	0	—	*b*	1840	3.7	68	1760	9	

a high naphthenic + aromatic
b The TM is very high and cannot be measured accurately
c Too high to measure

The data in this table have been used to derive equations for the variation in each property as a function of concentration. These equations can be used to calculate the value of the property concerned at any given concentration of ingredients or to plot a family of curves (given in Figures 1—9 of the accompanying drawings) showing the variation in that property as a function of concentration. Conversely, these equations and curves can be readily used to determine the concentrations of the ingredients to be blended to obtain any desired physical properties. Figures 1 to 9 show respectively the changes in: tensile modulus; stress at yield point; elongation at yield point; work (pounds/in); tensile strength, elongation at failure (%); Spencer impact; high load melt index and medium load melt index for compositions comprising various proportions aluminium silicate filler, mineral oil plasticizer and polyethylene of high load melt index 1.8. The Figures use triangular coordinates to express the various concentrations of the three ingredients.

The basic equation used to derive the curves is as follows: $Y_i = b_o + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2$, where $X_1 = \%$ oil, $X_2 = \%$ filler, and Y_i is the physical property. In order to obtain the curves, the coefficients b_o, b_1, b_2, b_{11}, b_{22} and b_{12} are solved by applying the data to the basic equation.

The actual application of the data and evaluation of coefficients is done in the following manner, using tensile modulus as the property to be evaluated in terms of filler and plasticiser concentration.

Consider the following experimental data:

Sample No.	4	12	13	14	15	22
Filler concentration (%)	0	20	20	30	30	40
Plasticiser concentration (%)	17.5	30	40	20	30	30
Tensile modulus p.s.i. 1000	65	87	66	139	119	139

Inserting these values into the basic equation, 6 equations are obtained so that the six coefficients may be obtained, as shown below. (Note that in deriving Figures 1—9, more than six equations have been used to evaluate the six coefficients. This leads to greater certainty in these values).

Basic: $Y_i = b_0 + b_1X_1 + b_2X_2 + b_2X_2 + b_{11}X_1 + b_{22}X_2 + b_{12}X_1X_2$

1) $65 = b_0 + 17.5b_1 + 0\ b_2 + 17.5^2b_{11} + 0^2b_{22} + (17.5 \times 0)b_{12}$

2) $87 = b_0 + 30\ b_1 + 20\ b_2 + 30^2\ b_{11} + 20^2\ b_{22} + (30 \times 20)\ b_{12}$

3) $66 = b_0 + 40\ b_1 + 20\ b_2 + 40^2\ b_{11} + 20^2\ b_{22} + (20 \times 40)b_{12}$

4) $139 = b_0 + 20\ b_1 + 30\ b_2 + 20^2\ b_{11} + 30^2\ b_{22} + (20 \times 30)b_{12}$

5) $119 = b_0 + 30\ b_1 + 30\ b_2 + 30^2\ b_{11} + 30^2\ b_{22} + (30 \times 30)b_{12}$

6) $139 = b_0 + 30\ b_1 + 40\ b_2 + 30^2\ b_{11} + 40^2\ b_{22} + (30 \times 40)b_{12}$

These equations are then solved simultaneously, and values for the coefficients are thus obtained. In order to set up curves such as are shown in Figure 1, arbitrary values of the tensile modulus are set up, for example 250,000. This value (divided by 1,000, for convenience) is then assigned as Y_i. A given filler concentration is then selected as X_2, such as 20%, and the basic equation is applied, inserting the experimentally determined values of coefficients, and X_1, plasticiser concentration, is solved for. In order to obtain different points along the same curve, the same value for TM is inserted and the filler concentration X_2 is varied, thereby giving different values for plasticiser concentration, X_1. Additional curves for the TM of the same system are obtained by inserting different evaluations for Y_i and following the same procedure.

In general, the curves in this illustration show that changing molecular weights have little effect on the values of TM, S_{yp}, and TS. However, increasing the molecular weight increases the values of E_{yp}, E_f, Work and SI, and decreases the MLMI. Further, it is apparent that less oil can me used with a

polymer of lower molecular weight for a given melt index, and that as a result the values of TM, S_{yp}, and TS would be higher, but the values of E_{yp}, E_t, SI and Work suffer, and with a much lower molecular weight (e.g. filled, plasticised conventional polyethylene), the products are too brittle to be useful. Generally, using polymer of much higher molecular weight improves the latter properties, but also increases the viscosity. Adding oil to lower the viscosity would decrease the TM, S_{yp}, and TS. Consequently, it becomes apparent that the optimum molecular weight will be different for different proposed use of the material.

Tensile Modulus, Figure 1.

Table II gives two sets of TM values. The "measured" values are those obtained for the actual samples by the testing laboratory and were used in computing the equation for the curves in Figure 1. The "computed" values are those calculated using this equation (or estimated from the curves) for samples of the same composition. The differences between the measured and computed values are quite small, showing the good fit of the curves to the data.

TABLE II

Tensile Modulus

Sample No. *a*	4	5	6	12	13	14	15	17	21	22	23
Measured TM p.s.i./1000	65	47	38	87	66	139	119	80	176	139	*b*
Computed TM p.s.i./1000	70*c*	60*c*	40*c*	86	63	145	116	84	177	139	203
Difference %	8	22	5	1	5	4	6	5	1	0	—

a. Numbers of Table I.
b. No value was obtained, but the sample seems quite stiff.
c. These values were estimated from the curves.

Samples 4, 5 and 6 were prepared with oil concentrations of 20, 30 and 40 per cent. After they had been tested, the samples were analysed for true oil content. Weighed portions were dissolved in hot xylene. When the solutions cooled the particulate polyethylene which precipitated out was washed, dried and weighed. The actual oil content is shown by the compositions listed in Table I.

This experiment shows that in the absence of filler, the oil (plasticiser) rapidly exudes from the polymer phase and is thereby lost.

Figure 1 shows that the TM is influenced mainly by the ratio of aluminium silicate to oil in the system. The polyethylene concentration has a much smaller effect.

Stress at Yield Point, Figure 2.

The measured and computed values of S_{yp} are compared in Table III. Again the values are in good agreement.

TABLE III

Stress at Yield Point

Sample No. *a*	4	5	6	12	13	14	15	17	21	22	23
Measured S_{yp} psi.	2270	1770	1430	1660	1160	2180	1610	1100	2270	1620	1840
Computed S_{yp} psi.	2250	1900	1350	1670	1160	2250	1640	1125	2130	1540	1930
Difference %	1	7	6	1	0	3	2	2	6	5	5

a. See Table I.

The S_{yp} is affected in a different manner from the TM by the concentration of the components. In this case the ratio of polyethylene to aluminium silicate has little effect, while

the amount of oil present has a very significant effect in lowering the value of S_{yp}.

Elongation at Yield Point, Figure 3.

Table IV gives the measured and computed E_{yp} values.

TABLE IV

Elongation at Yield Point

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured E_{yp} %	43.4	50.2	42.9	25.0	19.5	10.8	12.6	10.9	6.9	5.2	3.7
Computed E_{yp} %	42	43	44	21	19	13	12	11	6.8	6.2	3.3
Difference %	3	14	2	16	3	20	5	1	2	19	11

As Figure 3 shows, the aluminium silicate concentration as the greatest effect on the E_{yp} values. Changing the polyethylene/oil ratio at any aluminium silicate concentration has little effect.

Work, Figure 4

Again there is good agreement between measured and computed values (Table V). It is interesting that the effect of the oil in reducing the S_{yp} is almost balanced by the effect of the HR in reducing the E_{yp} so that the value of work is almost independent of the filler/oil ratio, increasing as the concentration of polyethylene increases (Figure 4).

TABLE V

Work

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured Work lb./in.	988	888	614	415	227	236	203	120	157	85	68
Computed Work lb./in.	950	850	650	350	224	285	196	120	145	95	64
Difference, %	4	5	6	16	1	20	3	0	8	13	6

Tensile Strength

Figure 5

The contours for TS are very similar to those of S_{yp}. The curvature in the opposite direction, particularly in the regions of high polyethylene concentration, reflects the greater effect of effective molecular weight on this property.

TABLE VI

Tensile Strength

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured TS, psi.	4300	3640	2560	1610	1540	1710	1410	963	2010	1400	1760
Computed TS, psi.	4000*a*	3200*a*	2400	1650	1310	1920	1410	1110	1180	1310	1840
Difference, %	7	12	6	2	15	12	0	16	41	6	5

a. Estimated

Elongation at Failure, Figure 6.

As is the case with the E_{yp} values, the aluminium silicate has the most significant effect. The values in the region of low aluminium silicate concentration and high polyethylene concentration are somewhat doubtful. No samples in this concentration range were used in computing the curves, however.

TABLE VII

Elongation at Failure

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured EF, %	1140	1250	1230	850	1190	175	54	68	38	63	9
Computed EF, &	950*a*	1200*a*	2000*a*	460	540	182	144	140	47	31	8
Difference, %	18	4	63	46	55	4	167	106	24	51	8

a. Estimated

Spencer Impact, Figure 7.

The curves of Figure 7 reflect the importance of molecular weight on SI. The measured and computed values agree quite well at intermediate concentrations (Table VIII). At high polyethylene levels the samples frequently did not fail, while at high aluminium silicate concentrations the samples were too brittle to test.

TABLE VIII

Spencer Impact

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured SI, psi.	*a*	*a*	.326	.253	.224	.230	.172	.148	.178	.126	*b*
Computed SI, psi.	.53*c*	.45*c*	.33*c*	.255	.211	.219	.182	.160	.135	.117	.07
Difference, %	—	—	1	1	6	5	6	8	24	7	—

a. Did not fail in test.
b. Too brittle to test.
c. Estimated.

High Load Melt Index and Medium Load Melt Index, Figures 8 and 9

TABLE IX

High Load and Medium Load Melt Index

Sample No.	4	5	6	12	13	14	15	17	21	22	23
Measured HLMI	2.3	6.6	16	5.5	43	1.9	8.6	59	.12	.10	*a*
Computed HLMI	7*b*	8*b*	50*b*	10	94	1.4	2.8	24	.16	.30	.008
Difference, %	200	21	210	82	120	26	68	59	36	200	—
Measured MLMI	.12	.45	1.1	.73	2.9	.10	.35	1.7	.002	*a*	*a*
Computed MLMI	.7*b*	.8*b*	3*b*	.51	5.7	.08	.09	.70	.008	.006	.0003
Difference, %	480	78	170	30	97	22	72	59	300	—	—
Measured HL/ML	19	15	15	7.5	15	19	25	35	60	—	—
Computed HL/ML	10	10	17	20	21	18	31	34	20	50	27

a. Unmeasurably low.
b. Estimated from curves.

Although the percentage differences between the measured and computed values of HLMI or MLMI are large, (Table IX), the values might be said to be in reasonable agreement considering the range of values covered. The curve for the HLMI (Figure 8) is very similar to that of the MLMI (Figure 9). In both cases the ratio of oil to polyethylene is the most important factor at low aluminium silicate concentrations, but at high aluminium silicate concentrations the effect of the filler concentration predominates. The values of HLMI and MLMI, measured or computed, generally differ by factors from 10 to 50 (HLMI/MLMI in Table IX). This ratio indicates the relative change in the apparent viscosity of the samples with shear rate, and might be called a "thixotropy index." The values appear to increase with both filler and oil concentration. Such behaviour indicates that some of these samples can be successfully moulded at high shear rates even though they have very low melt indices.

The operation of the invention is further clarified by considering a given system in which the only constant is the quantity of filler material, and in which the high molecular weight polyethylene is blended with GREX brand polyethylene, standard load melt index 0.7 (referred to as "low molecular weight polyethylene) as shown in Table X. Here, dibutyl sebacate (DBS) is used as the plasticiser, and calcium carbonate + 2.5% C_5—C_{20} fatty acid is the filler material. This illustration demonstrates the effects of polymer molecular weight and plasticiser concentration on physical properties.

As in the previous illustration, the data in this table have been used to derive equations for the variation in each property as a function of concentration. Figures 10 to 17 show the curves obtained respectively showing variations in tensile modulus; stress at yield point; elongation at yield point; work (pound/in); tensile strength; elongation at failure (%); Spencer Impact and medium load melt index.

TABLE X

Properties *a* of 30% by Volume Calcium Carbonate + 2.5% Fatty acid Blends.

Sample No.	Composition, Percent by Volume: 0.7 SLMI Polyethylene	0.01 HLML Polyethylene	DBS	TM, psi	Syp, psi	Eyp, %	Work in. ×psi	TS	EF	SI	MLMI
24	70	0	0	371,000	—	—	(26)*b*	2200	1.2	brittle *c*	.54
25	63	7	0	335,000	—	—	(42)*b*	2620	1.6	brittle *c*	.14
26	56	7	7	268,000	—	—	(47)*b*	2140	2.2	brittle *c*	.54
27	49	21	0	292,000	—	—	(55)*b*	2900	1.9	.015	.002
28	42	21	7	218,000	2760	3.2	88	2480	17	.12	.01
29	42	14	14	163,000	1840	3.3	60	1600	5.3	.09	.16
30	42	7	21	119,000	1310	2.7	35	1300	3.5	—	3.9
31	35	35	0	294,000	3340	2.2	74	2950	287	no failure *e*	—
32	35	21	14	153,000	2020	3.6	73	1490	175	.27	.05
33	35	14	21	119,000	1420	3.3	47	1050	208	.19	.32
34	28	35	7	271,000	2590	3.3	86	2160	208	no failure *e*	0
35	28	21	21	114,000	1300	3.0	39	1070	348	.19	.25
36	28	7	35	54,000	560	2.7	15	490	227	.05	25.
37	14	28	28	68,000	900	3.8	34	1120	640	.30	.16
38	100			243,000	4090	9.2	380	2830	880	.185	— (0.7)*e*
39	80	20		193,000	3860	9.5	367	2840	322	.226	— (0.6)*e*

a TM = tensile modulus, SYP = stress at yield point, EYP = elongation at yield point, Work = SYP × EYP (inches) /2, TS = tensile strength, EF = elongation at failure, SI =Spencer impact, MLMI = medium load melt index (7840 g.)

b Work = TS × EF/2

c Could not be clamped in tester.

Tensile Modulus, Figure 10

The measured and computed values of TM are compared in Table XI. The differences between the measured and computed values are generally quite small. The average percentage difference between the observed and computed values is less than the average standard deviation (as percent) reported by the testing laboratory for these samples.

TABLE XI

Tensile Modulus

Sample No. *a*	24	25	26	27	28	29	30	32	33	35	37	A
Measured TM p.s.i./1000	371	335	268	292	218	163	119	153	119	114	68	
Computed TM p.s.i./1000	396	331	256	297	223	165	134	159	110	104	73	
Difference, %*b*	6.7	1.2	4.5	1.7	2.3	1.2	13	3.9	7.5	8.8	7.3	5
Std. Deviation, %*b*	5.1	6.6	3.4	6.5	5.5	4.9	5.9	11	5.9	2.6	5.9	5

a Numbers from Table X. Values for TM were not computed for samples 8, 11, and 13. The experimental values for samples 8, 11 and 13 were used in computing the equation.

b As percent of the measured TM.

Inspection of Figure 10 shows that the TM in this system is reduced markedly by increasing DBS concentrations at any ratio of low molecular weight to high molecular weight polyethylene. It is noteworthy that there is an initial decrease and then an increase in TM as high molecular weight polyethylene is substituted for conventional polyethylene at a constant DBS concentration. This is equivalent to using polyethylene of uniformly increasing molecular weight.

Stress at Yield Point, Figure 11.

The measured and computed values of S_{yp} are compared in Table XII.

TABLE XII

Stress at Yield Point

Sample No.	24	25	26	27	28	29	30	32	33	35	37	A
Measured SYP, psi.	2200*a*	2620*a*	2140*a*	2900*a*	2760	1840	1310	2020	1420	1300	900	
Computed SYP, psi.	2340	2630	2210	3010	2470	1900	1380	1940	1440	1440	770	
Difference, %	6.4	0.4	3.3	3.8	11	3.3	5.4	4.0	1.4	4.6	15	5
Std. Deviation, %	9.5	4.5	8.1	7.5	2.1	3.2	4.4	1.4	1.6	1.8	2.5	4

a Values of Tensile Strength were used for these samples in the computation since no yield point was found on the stress strain curves.

Again, the values are in good agreement.

The S_{yp} is also decreased by increasing concentrations of DBS at any ratio of low molecular weight to high molecular weight polyethylene (Figure 2). An increase in S_{yp} with an increase in the ratio at constant DBS concentration is noted, particularly at low DBS levels. This change is much less marked than that caused by varying the DBS concentration.

Elongation at Yield Point Figure 12.

Table XIII shows the measured and computed values of E_{yp}.

TABLE XIII

Elongation at Yield Point

Sample No.	24	25	26	27	28	29	30	31	32	33	34	35	36	37	Ave.
Measured EYP, %	1.2*a*	1.6*a*	2.2*a*	1.9*a*	3.2	3.3	2.7	2.2	3.6	3.3	3.3	3.0	2.7	3.8	
Computed EYP, %	1.4	1.5	2.4	1.9	2.7	3.1	3.1	2.1	3.3	3.3	3.0	3.5	2.6	3.7	
Difference, %	17	6.3	9.1	0	16	6.1	15	4.6	8.3	0	9.1	17	3.7	2.6	8.2
Std. Deviation, %	8.3	0	18	16	3.1	6.1	0	4.6	8.3	3.0	12	3.3	7.4	2.6	6.6

a Values of EF were used when no yield point was observed.

At any ratio of low molecular weight to high molecular weight polyethylene the E_{yp} increases to a maximum and then decreases with increasing DBS concentrations (Figure 3). It is interesting that this maximum occurs at an almost constant (21% by volume) DBS concentration. At any given DBS concentration, increasing the said ratio increases the E_{yp}.

Work, Figure 13.

There are considerable differences between the measured and computed values of work, as shown by Table XIV.

TABLE XIV

Work

Sample No.	1	2	3	4	5	6	7	9	10	12	14
Measured Work, p/in.	26*a*	42*a*	47*a*	55*a*	88	60	35	73	47	39	34
Computed Work, p/in.	20	38	45	70	71	57	40	66	48	54	37
Difference, %	23	9.5	4.3	27	19	5.0	14	9.6	2.1	39	8.9

a $\frac{1}{2} \times TS \times EF$ was used in these cases since there were no S_{yp} and E_{yp} values.

Since the work is a product of the S_{yp} and E_{yp}, the standard deviation would be greater than that of those properties.

The contours of the work function reflect the influence of the S_{yp} and E_{yp}. At low ratios of low molecular weight to high molecular weight polyethylene and low DBS concentration the relative changes in E_{yp} (increasing) with increasing DBS concentration are greater than the corresponding relative changes in S_{yp} (decreasing), and therefore the work increases. As the E_{yp} levels off, the changes in S_{yp} predominate and the work decreases with increasing DBS concentrations. When both the E_{yp} and S_{yp} decrease, the work decreases at a more rapid rate. At higher ratios of low molecular weight to high molecular weight polyethylene (but not at constant low molecular weight polyethylene concentrations) the decrease in S_{yp} predominates initially and the work decreases at a slow rate.

Tensile Strength, Figure 14.

The contours for TS are very similar to those of S_{yp}. The decrease in TS with increasing DBS concentration is somewhat greater than the decrease in S_{yp}.

TABLE XV

Tensile Strength

No.	24	25	26	27	28	29	30	31	32	33	34	35	36	37
Meas.	2200	2620	2140	2900	2480	1600	1300	2950	1490	1050	2160	1070	490	1120
Comp.	2400	2620	1990	2900	2250	1630	1060	2900	1700	1200	2210	1260	570	910
Diff.	9.1	0	7.0	0	9.3	1.9	19	1.7	14	14	2.3	18	16	19
Std. Dev.	9.5	4.5	8.1	7.2	9.2	11	4.3	1.7	3.0	2.0	11	3.4	1.0	3.0

Elongation at Failure, Figure 15.
The values in Table XVI show the trend illustrated in Figure 6.

TABLE XVI

Elongation at Failure

Sample No.	24	25	26	27	28	29	30	32	33	35	37	Ave.
Measured EF, %	1.2	1.6	2.2	1.9	17	5.3	3.5	175	208	348	640	
Computed EF, %	0.6	1.1	2.7	5.3	16	17	18	50	49	160	2240	
Difference, %	50	31	23	180	5.9	230	340	71	73	54	250	120
Std. Deviat., %	8	0	18	16	77	6	3	30	9	15	4	26

These values indicate that the EF is independent of the ratio of high molecular weight polyethylene to DBS and increase with decreasing conventional (low molecular weight) polyethylene concentration. It is noted that separately or combined, high molecular weight polyethylene and DBS are equally efficient in increasing the EF.

Spencer Impact, Figure 16.
The contours of Figure 16 are based on more limited data than the rest of the charts in this series. However, the curves illustrate the trends at the different concentrations of the components.

Medium Load Melt Index, Figure 17.
Even though the differences between the measured and computed MLMI values are large (Table XVII), they may be said to be in very good agreement when it is considered that the values listed cover a range of more than 10^4 (.002 to 25).

TABLE XVII

Medium Load Melt Index

Sample No.	24	25	26	27	28	29	30	32	33	35	37	Ave.
Measured MLMI	.54	.14	.54	.002	.01	.16	3.9	.05	.32	.25	.16	
Computed MLMI	.94	.13	.38	.002	.01	.22	3.3	.04	.75	.17	.19	
Difference, %	74	7	30	0	0	38	15	20	125	32	19	33

In general it can be said that increasing the concentration of high molecular weight polyethylene decreases the melt index (independently of the ratio of the low molecular weight polyethylene to DBS) and that increasing the concentration of DBS increases the melt index (independent of the ratio of low molecular weight to high molecular weight polyethylene). It appears that the effects are equivalent at a ratio of DBS to high molecular weight polyethylene of approximately 55 to 45. That is, the MLMI does not vary from approximately 1 as the concentration of low molecular weight polyethylene is changed.

A still further illustration of the invention is provided by a series of samples containing 30% by volume calcium carbonate + 2.5% fatty acid and varying concentrations of conventional polyethylene (0.7 SL melt index), high molecular weight particulate polyethylene (1.8 HL melt index) and paraffin oil (335—350 viscosity). See Table XVIII.

TABLE XVIII

Properties of compositions comprising 30% by Volume Fatty Acid-coated calcium carbonate

No.	Composition, % by volume			Tm psi	SYP psi	Eyp %	Work p/in	TS psi	EF %	SI psi
	0.7 SLMI Polyethylene	1.8 HLMI Polyethylene	Oil							
40	63	0	7	200000	—	—	(50)*a*	1990	2.5	—
41	49	21	0	336000	—	—	(61)*a*	3030	2.0	—
42	49	0	21	105000	—	—	(48)*a*	1320	3.6	—
43	35	35	0	297000	—	—	(88)*a*	3260	2.7	.03
44	28	35	7	182000	2410	3.6	87	2330	4.6	.05
45	28	21	21	92000	1400	4.5	63	1310	12	.11
46	14	35	21	87000	1450	5.7	83	1080	277	.18
47	0	49	21	89000	1410	5.2	73	1300	523	.25
48	0	35	35	49000	760	5.0	38	830	816	.21

a Work = EF × TS × 1/2

Families of curves computed from these data are very similar in shape to those computed for the corresponding properties in the previous illustration. Figures 18 to 24 show the curves obtained respectively showing variations in tensile modulus; stress at yield point; work (pound/in) tensile strength; elongation at failure (%); Spencer impact and medium load melt index.

The tensile modulus curves (computed from the data given for samples 40 to 48, Figure 18) show much less curvature than the curves in Figure 10. This is due to the lower molecular weight range of polyethylene. Equivalent values of TM are found for comparable compositions in the two series.

The S_{yp} (Figure 19) and TS (Figure 21) curves coincide quite closely in shape and value with those of Figures 11 and 14.

The curves for EF (Figure 22) curve more than those in Figure 15, but the same trend is shown. The same is true for the SI curves (Figure 23). The MLMI (Figure 24) shows the same trend as Figure 17 but the changes are much smaller in magnitude. The particulate 1.8 HL melt index polyethylene has a smaller effect than the higher molecular weight 0.1 HL melt index polyethylene.

The contour diagrams in the foregoing illustrations can be readily used to select a composition which will provide desired physical properties. A simple way of utilising them is to prepare the curves on transparent films. The relationship between any of the properties can then be seen when the transparent overlays are superimposed on each other. To illustrate this, a material having a S_{yp} of at least 1500 p.s.i. an EF greater than 100% and a minimum MLMI of .01 might be desired, in a blend consisting of calcium carbonate + 2.5% fatty acid DBS, conventional polyethylene, and high molecular weight polyethylene. When the curves for S_{yp} (Figure 11), EF (Figure 15) and MLMI (Figure 16) are superimposed, the above limits define a range of concentrations. Further, the range of properties available in this concentration range can be read from overlays of any of the other properties.

The breadth and scope of the invention can further be seen by a consideration of Tables XIX—XXIX, which give some of the numerous filler, plasticiser, and polymer combinations which have been tested and which have proved to be satisfactory.

Several special tests have been run on some of the samples heretofore described. The results of these tests are given in Table XXX.

TABLE XIX

Comparison of Samples Containing 0.01 HLMI Polyethylene, 0.7 SLMI Polyethylene, 30% by Volume Filler, and 21% Plasticiser

A. Filler: $CaCO_3$, fatty acid coated

Sample No.	Plasticiser	Polymer %0.01 HLMI PE	Polymer %0.7 SLMI PE	T.M. (p.s.i.)	Syp (p.s.i.)	Eyp. %	Work p/in.	TS (p.s.i.)	EF. %	SI (psi)	Melt Index ML	Melt Index HL
49	none-control	21	49	292,000	—	—	55	2900	1.9	0.08	0.002	
50	Polyisobutylene	7	42	123,000	1390	4.2	58	1380	4.7	—	0.22	
51	Polyisobutylene	21	28	123,000	1590	6.2	99	1260	390	0.18	0.02	
52	Polyisobutylene (7%)	21	42	227,000	2760	2.9	80	2420	3.8	0.06	0.002	
53	paraffin wax, m.p. 48°C.	21	28	180,000	2330	5.6	130	1650	131	0.04	0.02	
54	heavy mineral oil (viscosity = 335—350 SSU at 100°F.)	21	28	90,000	1550	6.4	99	1530	478	0.36	0.02	0.16
55	light mineral oil (viscosity =125—135)	21	28	108,000	1610	5.4	87	1430	3.42	0.19	0.016	0.70
56	Dioctyl Sebacate	21	28	92,000	1430	4.4	63	1270	399	0.33	0.20	0.80
57	Octyl epoxy tallate	21	28	104,000	1450	3.6	52	1360	389	0.30	0.09	
58	Polyester glycol	21	28	177,000	1530	2.2	34	1540	199	0.24	0.02	
59	Chlorinated Biphenyl	21	28	147,000	1950	4.0	78	1530	9	0.21	0.004	
60	Ethylene/vinyl acetate copolymer	21	28	145,000	2460	7.5	184	2130	41	0.06	0.01	0.23

B. Plasticizer: dibutyl sebacate

Sample No.	Filler											
61	Precipitated Calcium Carbonate (0.1 to 0.35 μ)	21	28	108,000	1800	6.6	120	1290	178	0.14	0.22	5.5
62*b*	Kaolin (34%)	16	36	153,000	2630	13.9	366	2260	41	—	0.06	1.7
63*c*	Kaolin (34%)	24	20	101,000	1780	10.6	188	1710	137	0.30	0	0.68
64	Precipitated Calcium carbonate (0.03—0.04 μ)	21	28	137,000	2060	5.8	119	1740	48	0.14	0.002	
65*c*	Hydrophobic kaolin (34%)	24	20	114,000	1810	12.2	221	1610	46	0.16	0.01	

a —Work = TSX EF/2 × 1.27 *b* — 15% Plasticizer *c* — 23% Plasticizer

TABLE XX

Comparison of Samples containing 0.01 HLMI Polyethylene, 0.7 SLMI Polyethylene, 30% by volume filler, and plasticizer.

Sample No.	Plasticizer	Filler	Polymer % 1.8 HLMI PE	Polymer % 0.7 SLMI PE	TM (p.s.i.)	Syp (p.s.i.)	Eyp %	Work p/in.	TS (p.s.i.)	EF. %	SI (psi.)	Melt Index ML	Melt Index HL
66	polyisobutylene (21%)	precipitated calcium carbonate	21	28	97,000	1420	510	71	1100	12	0.05	0.12	
67	polyisobutylene (21%)	precipitated calcium carbonate	35	14	81,000	1210	4.1	50	900	154	0.16	0.14	3.1
68	polyisobutylene (35%)	precipitated calcium carbonate	21	14	56,000	850	6.7	57	580	200	0.07	0.15	39
69	Dibutyl sebacate (21%)	fatty acid coated calcium carbonate	21	28	119,000	1400	3.5	49	1330	18	0.15	1.1	29
70	Dibutyl sebacate (7%)	fatty acid coated calcium carbonate	35	28	204,000	2430	3.6	88	2310	9.1	.07	.13	2.7

TABLE XXI

Physical Properties of Samples Containing 30% by volume filler with 30% by volume paraffin oil (viscosity 335—350 SSU at 37.8°C.) in 1.8 HLMI Polyethylene.

No.	Filler	Melt Index SL	Melt Index ML	Melt Index HL	Thio. index	Tensile Modulus psi.	Stress at Yield point psi.	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi.	Elongation at Failure %	Spencer Impact psi.
71	kaolin	0	.34	9.0	27	141000	1640	15.2	250	1310	106	.181
72	organophilic kaolin	0	.32	8.4	26	119000	1570	16.6	261	1280	359	.173
73	calcium silicate, natural	.095	.88	18	21	121000	1350	10.5	142	1220	29	.035
74	calcium silicate, precipitated	0	0	0	—	200000	1900	4.2	80	1630	33	brittle
75	muscovite mica	.064	.82	13	16	136000	1570	7.7	121	1510	9	.030
76	calcium carbonate, precipitated	0	.06	4.3	72	63000	980	2.7	39	1150	4	brittle
77	calcium carbonate, fatty acid coated	0	0	1.3	—	86000	1020	5.5	56	870	590	.209
78	calcium carbonate, organophilic	.09	1.5	22	14	45000	920	12.8	118	1190	1030	.188
79	calcium carbonate, natural (chalk)	0.6	.65	9.0	14	82000	860	8.7	75	910	770	.100
80	magnesium oxide	0	0	0	—	170000	—	—	(41)[a]	1520	3	.019
81	magnesium carbonate	0	0	0	—	970000	900	4.2	47	860	16	—[f]
82	stannic oxide	.026	.69	21	30	151000	1700	27.3	464	1170	72	.117
83	glass beads (400 mesh)	.028	.89	13	15	82000	770	4.8	37	740	470	.081
84	carbon black, thermal	.008	.30	6.5	22	65000	1500	44.5	668	1470	512	.227
85	carbon black, channel	0	0	0	—	172000	1910	10.5	201	1720	142	.120
86	carbon black, furnace	0	0	0	—	128000	1230	3.2	39	540	7	—[f]

[a] $= \frac{1}{2} \times TS \times EF$

TABLE XXII

Physical Properties of Samples Containing 30% by Volume Filler with 30% by volume oil (High naphthenic + aromatic, viscosity 559 SSU at 37.8°C.) in 1.8 HLMI polyethylene.

No.	Filler	Melt Index SL	ML	HL	Thixo. index	Tensile Modulus psi.	Stress at Yield point psi.	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi.	Elongation at Failure %	Spencer Impact psi.
87	kaolin	.008	.33	6.3	19	127000	1520	14.2	216	1440	34	.114
88	"polysilicate of alumina", United Clay Mines Co.	.069	1.0	14	14	66000	990	12.5	124	830	390	.067
89	montmorillonite, organophilic	.002	.01	1.5	150	70000	1020	28.2	273	970	52	.050
90	attapulgite	0	0	.12	—	—	2100	3.4	72	2060	9	.054
91	fibrous talc	.046	.82	16	20	179000	1550	8.3	129	1400	50	.080
92	chrysotile asbestos (shorts)	0	0	.20	—	119000	1220	6.4	78	1100	11	.043
93	chrysotile asbestos (floats)	0	.04	3.2	80	96000	1130	10.1	114	940	325	.087
94	chrysotile asbestos (fiber)	0	0	0	—	151000	1440	2.7	39	1150	4	brittle
95	glass beads (400 mesh)	.083	1.3	16	12	48000	970	36.4	353	1120	780	.095
96	glass flake	.016	.61	19	31	83000	660	17.3	114	340	21	.045
97	wood flock	.066	1.1	21	19	65000	930	22.8	212	830	160	.061
98	27% organophilic calcium carbonate + 3% silica gel	.048	.85	12	14	52000	1060	14.3	152	1050	820	.183
99	27% fatty acid coated calcium carbonate + 3% magnesium oxide	0	.02	.63	32	127000	1240	4.7	58	1070	20	.122
100	27% precipitated calcium carbonate + 3% magnesium oxide	.004	.18	3.7	21	114000	1250	7.0	88	1040	29	.118

TABLE XXIII

Physical Properties of Samples Containing 30% by Volume Filler and 30% by Volume Dioctyl Sebacate Plasticizer in 1.8 HLMI Polyethylene.

No.	Filler	Melt Index			Thixo. index.	Tensile Modulus psi.	Stress at Yield point psi.	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi.	Elongation at Failure %	Spencer Impact psi
		SL	ML	HL								
101	kaolin	.018	.23	6.9	30	76000	1100	11.5	127	990	91	.117
102	kaolin, organophilic	0	.36	9.7	27	78000	1290	12.8	165	1150	84	.192
103	calcium silicate, natural	.12	2.0	32	16	71000	990	14.6	144	880	204	.089
104	chrysotile asbestos (fiber)	0	0	0	—	84000	910	3.7	34	720	5	.032
105	calcium carbonate, precipitated	.007	.45	9.4	21	80000	910	5.7	52	820	460	.183
106	calcium carbonate, fatty acid coated	.016	.49	11	23	101000	860	4.4	38	920	490	.222
107	calcium carbonate, organophilic	.14	2.0	27	14	76000	900	12.2	110	1260	1050	.220
108	calcium carbonate, natural (chalk)	.094	1.3	21	16	84000	880	10.1	89	730	210	.096
109	silica gel (0.022 μ)	0	0	.08	—	83000	1230	4.3	53	1030	315	.174
110	stannic oxide	.066	2.4	43	18	97000	1080	7.7	83	980	440	.252
111	glass beads (400 mesh)	.10	1.1	17	15	61000	770	13.2	102	730	390	.070
112	carbon black, thermal	0	.41	8.3	20	106000	1560	37.6	587	1420	560	.234
113	carbon black, channel	0	0	0	—	134000	1720	20.7	356	1630	350	.156
114	carbon black, furnace	0	0	0	—	134000	1250	3.8	48	880	6	brittle

TABLE XXIV

Physical Properties of Samples containing 30% by Volume Filler and 30% by Volume Polyisobutylene Plasticizer in 40% 1.8 HLMI Polyethylene.

No.	Filler	Melt Index SL	Melt Index ML	Melt Index HL	Tensile Modulus psi	Stress at Yield Point psi	Elongation at Yield Point %	Work Pounds/ inch	Tensile Strength psi	Elongation at Failure %	Spencer Impact psi
115	kaolin	0	.04	1.7	241000	2130	6.6	140	1900	19	.147
116	kaolin organophilic	0	.21	5.9	122000	1470	14.9	220	1230	133	.170
117	calcium silicate, natural	.083	.95	17	121000	1130	7.3	83	960	90	.049
118	calcium carbonate, precipitated	0	.05	4.3	103000	960	6.5	62	820	530	.139
119	calcium carbonate, fatty acid coated	0	0	.36	92000	1000	6.2	62	820	430	.111
120	calcium carbonate, organophilic	.13	2.2	36	57000	850	10.6	90	1120	1095	.146
121*	calcium carbonate, natural (chalk)	.054	.84	12	95000	810	36.1	293	840	546	.095
122*	stannic oxide	.043	1.0	16	126000	1520	20.3	308	1090	242	.138
123*	glass beads (400 mesh)	.027	.57	10	83000	770	33.7	258	770	423	.083
124	calcium carbonate, precipitated		.03	5.1	45000	790	6.7	52	460	131	.10

* Polyisobutylene of a lower molecular weight was used.

TABLE XXV

Physical Properties of Samples containing 30% by Volume Kaolin and 30% by Volume Plasticizer in 40% 1.8 HLMI Polyethylene.

No.	Plasticiser	Melt Index			Thixotropy Index	Tensile Modulus psi	Stress at Yield Point psi	Elongation at Yield Point, %	Work Pounds/inch	Tensile Strength psi	Elongation at Failure %	Spencer Impact psi
		SL	ML	HL								
125	Paraffin oil (viscosity 75 SSU at 37.8°C.	.003	.26	5.0	19	—	2040	15.8	322	1590	270	.157
126	Paraffin oil (viscosity 256 SSU at 37.8°C.	0	.30	7.4	25	149000	1580	16.5	260	1320	290	.145
127	Paraffin oil (viscosity 58 SSU at 37.8°C.	.006	.36	9.0	25	184000	1800	14.1	254	1460	66	.123
128	Paraffin oil (viscosity 2110 SSU at 37.8°C.	0	.28	9.6	34	101000	1410	16.8	237	1220	550	.145
129	dilauryl ketone	.003	.24	12	50	391000	2440	9.4	230	2140	17	
130	asphalt	.010	.33	6.7	20	196000	1650	11.8	195	1190	630	.116
131	Dioctyl fumarate	.010	.32	5.6	18	94000	1340	12.0	161	1150	670	.181
132	Butyl stearate	.022	.58	15	26	90000	1260	12.4	156	1025	646	.181
133	Polyisobutylene	0	.04	1.7	43	241000	2130	6.6	140	1900	19	.147
134	Diisodecyl phthalate	.010	.67	13	20	81000	1130	10.8	122	960	196	.176

TABLE XXVI
Physical Properties of Miscellaneous Samples

No.	Composition, Percent by Volume			Melt Index		Tensile Modulus psi	Stress at Yield Point psi.	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi.	Elongation at Failure %	Spencer Impact psi
	Polymer	Filler	Plasticiser	ML	HL							
135	1.8 HLMI Polyethylene 50	kaolin 40	Dioctyl Sebacate 10	0	.047	285000	2690	5.9	159	2500	7	.186
136	0.4 HLMI particle form polyethylene 30	kaolin 30	Petroleum Oil* 40	.03	5.1	60000	890	41.	366	990	840	.177
137	0.01 HLMI polyethylene 30	kaolin 30	Petroleum Oil* 40	.002	.41	49000	—	—	—	1050	220	.273
138	1.8 HLMI polyethylene 30	kaolin 30	Dioctyl Sebacate 40	.51	5.1	56000	680	10.9	74	590	380	.130

* High naphthenic and aromatic petroleum oil, viscosity 559 in SSU at 37.8°C.

TABLE XXVII

Physical Properties of Samples Containing Water Soluble Plasticizers

No.	Composition, % by Volume			Melt Index		Tensile Modulus psi	Tensile Strength psi	Elongation at failure %	Spencer Impact psi
	Polymer	Filler	Plasticizer	ML	HL				
139	(1) PF 40	(2) Hydrite-R 30	Glycerol 30	.030	19	61,000	580	6.5	—
140	PF 40	Hydrite-R 30	ethylene glycol 30	0	.11	—	1120	26	—
141	PF 40	Hydrite-R 30	diethylene glycol 30	.008	2.1	123,000	880	24	—
142	PF 40	Hydrite-R 30	polyethylene glycol (M.W.-400) 30	.068	314	28,000	300	2.9	0.27

(1) High density polyethylene, O M.I.
(2) Kaolin clay

TABLE XXVIII

Physical Properties of Samples Containing Water Soluble and Water Insoluble Plasticizers

No.	Composition, % by volume				Melt Index		Tensile Modulus psi	Stress at Yield point psi	Elongation at Yield Point, %	Work Pounds/ inch	Tensile Strength psi	Elongation at Failure %
	Polymer	Filler	Plasticizer		ML	HL						
143	HiFax 1901 (1) 17	"HiSil" (2) 18	Glycerol 44	oil (3) SF412 21	0	.001	1210	—	—	—	84	36
144	17	18	44	21	—	—	8470	312	11.9	37	309	85.5

(1) Hercules polyethylene, O M.I., 15—16 RSV.
(2) "HiSil" is a Registered Trade Mark for finely ground silica.
(3) Fractionated petroleum oil.

TABLE XXIX

Physical Properties of samples Containing Water Soluble Fillers

No.	Composition, % by volume			Melt Index		Tensile Modulus psi	Stress at Yield Point psi	Elongation at yield point, %	Work Pounds inch	Tensile strength psi	Elongation at Failure %
	Polymer	Filler	Plasticizer	ML	HL						
145	PF (1) 50	NaCl 30	Oil 20	.22	3.6	94,000	1100	15.6	171	1200	610
146	40	KCL 30	Oil 30	6.9	too high to measure accurately	49,650	673	25.7	173	649	54.5
147	40	KCL 30	PPG (2) 30	1.8	96	62,890	755	6.43	48.5	636	9.42

(1) High density polyethylene, O M.I.
(2) Polypropylene glycol, molecular weight — 1200.

TABLE XXX

Special Properties

Sample No.	Refer to Table #	Tension Impact # ft/in²	Brittle Temperature, °C.	Vicat Softening Point	Hardness Shore D	Abrasion Resistance mg/1000 cyc.	Oxygen Permeability cc mil/atm M^2 day
18	I	17.7	—21.4	86.5	42		$8.23X10^3$
19	I			93.3	45		$7.9X10^3$
20	I			102	46		$8.4X10^3$
135	XXVI			94	44		$8.48X10^3$
136	XXVI	13.6		99.5	46		$10.05X10^3$
16	I					39.2	
137	XXVI	14.1	—11.2	88	39		$26.4X10^3$
100	XXIII	20.5	27.2	119.5	52	67.3	
134	XXVI	6.5		129.7	69	23.1	
1	I	118		130	64		$2.32X10^3$

In the Tension Impact test a dumbbell shaped sample was subjected to a sudden stress along its axis by a falling pendulum and the force required to rupture the sample was measured. Although these samples fall short of pure high molecular weight polyethylene (Sample 1) most are quite good as compared to some other moulding resins. For example, normal polypropylene generally has values from 10 to 30.

Three of the samples were tested for their brittleness temperature, i.e. the temperature at which half of the samples fail when they are flexed abruptly. The results of these tests are given in the table.

In general, it was found that the softening point and also the hardness of these materials decrease as the amount of plasticiser is increased.

The abrasion resistance of the few samples tested was less than that of conventional polyethylene but much higher than that of a sample of vinyl asbestos floor tile, which had a value of 134 mg/1000 cycles. The test consisted of weighing the sample before and after it was subjected to 1000 cycles of an abasive wheel. The weight loss in milligrams is a measure of abrasion resistance.

The oxygen permeability of all of the filled, plasticised samples was greater than that of high molecular weight, particulate polyethylene.

The compositions of the invention can be processed by extrusion, injection moulding, vacuum forming, or calendering. The very high thixotropy of many of these compositions enables them to be injection moulded quite readily. This thixotropy also prevents "snap-back" of items after they are vacuum formed, and prevents sag in extruded pipe while it is still hot.

Consideration only of the melt indices of the compositions would seem to indicate that they would be hard to process. However, as is indicated by the high thixotropy, an increase in the pressure on the compositions causing an increased rate of shear results in a substantial decrease in their viscosity so that their mouldability compares favourably with that of conventional polyethylenes.

Cold pressing produces additional changes in properties that are desirable under some circumstances. Specifically, the tensile modulus is lowered, while the elongation at yield is increased manyfold. X-ray analysis indicates that the crystallinity is reduced markedly by this process.

The compositions are useful for a great number of applications, for example low cost moulding resin for toys, etc., low cost films to cover seed beds, line irrigation ditches, liners for roofing and sliding materials, and self lubricating bearings for applications not requiring excessive strength. Further, as already stated, they can be used to prepare porous films and porous moulded items, inasmuch as some or all of the plasticiser, filler or both can be extracted with organic solvents, or, preferably, with water. To illustrate the extraction of plasticiser using an organic solvent, Sample 138 (Table XXVI) having initially an oxygen permeability of 26.4×10^3 was extracted with methanol for one hour at room temperature. During this extraction about 95% of the plasticiser was removed from the sample. The porous film thereby obtained had a greatly increased permeability to oxygen of $8,451 \times 10^3$ cc mil/Atm. M^2 day.

WHAT WE CLAIM IS:—

1. A composition comprising 5 to 90% of a polyolefine having a standard load melt index of substantially zero, 5 to 90% of inert filler material, and 5 to 90% of plasticizer (as hereinbefore defined), all percentages being by volume.

2. A composition according to claim 1, wherein the said polyolefine is polyethylene.

3. A composition according to claim 1 or 2, wherein the polyolefine of zero melt index is a polyethylene of density 0.93 to 0.97, a high load melt index of at most 1.8, and a viscosity of at least 4.0 measured on a solution of 0.02 gram of polymer in 100 grams of decalin at 130°C.

4. A composition according to claim 3, wherein the polyethylene has a high load melt index of 1.8 and a viscosity of 4.0.

5. A composition according to claim 3, wherein the polyethylene has a high load melt index of 0.01 to 1.8 and a viscosity of 9.3 to 4.0.

6. A composition according to claim 3, wherein the polyethylene has a high load melt index of 0.01 and a viscosity of 9.3.

7. A composition according to any one of claims 2 to 6, containing in addition 1—80% of polyethylene of standard load melt index 0.01 or higher.

8. A composition according to claim 7, wherein said polyethylene has a standard load melt index of 0.7 and a viscosity of 2.2 measured on a solution of 0.1 gram of polymer in 100 grams of decalin at 130°C.

9. A composition according to claim 8, comprising 5—85% of polyethylene of zero standard load melt index, density 0.93 to 0.97, and a high load melt index of 1.8; 5 to 40% of polyethylene of standard load melt index 0.7; 5 to 85% of inert filler material; and 5 to 85% of plasticizer.

10. A composition according to claim 8, comprising 5 to 85% of polyethylene of zero standard load melt index, density 0.93 to 0.97, and a high load melt index of 0.01; 5 to 75% of polyethylene of standard load melt index 0.7; 5 to 85% of inert filler material; and 5 to 85% of plasticizer.

11. A composition according to any one of the preceding claims, wherein the inert filler

comprises a material which is insoluble in water.

12. A composition according to claim 11, wherein the inert filler is one or more of the following: kaolin, calcium silicate, calcium carbonate, magnesium carbonate, magnesium oxide, stannic oxide, mica, glass beads, glass flake, asbestos, carbon black, silica, aluminium polysilicate, montmorillonite, attapulgit, talc and wood flock.

13. A composition according to any one of claims 1 to 10, wherein the inert filler comprises a material which is soluble in water.

14. A composition according to claim 13, wherein the inert filler is one or more of the following: sodium chloride, potassium chloride, calcium chloride, sodium acetate, potassium acetate, calcium acetate, copper acetate, barium acetate, sodium sulphate, potassium sulphate, sodium phosphate, potassium phosphate, sodium nitrate, potassium nitrate and sugar.

15. A composition according to any one of the preceding claims, wherein the plasticizer comprises a material which is insoluble in water.

16. A composition according to claim 15, wherein the plasticizer is one or more of the following: paraffin oil, paraffin wax, butyl stearate, dibutyl sebacate, dioctyl sebacate, ethylene/vinyl acetate copolymer, polyisobutylene, diisodecyl phthalate, dioctyl fumarate, asphalt, polyester glycol, octyl epoxy tallate, and chlorinated biphenyl.

17. A composition according to any one of claims 1 to 14, wherein the plasticizer comprises a material which is soluble in water.

18. A composition according to claim 17, wherein the plasticizer is one or more of the following: glycol, glycol ethers and esters, glycerin, glycerol monoacetate, diethylene glycol, diethylene glycol ethers and esters, triethylene glycol, polyethylene glycol (molecular weight of 400 to 20,000), propylene glycol, dipropylene glycol, polypropylene glycol (molecular weight of 260 to 1200), trimethylene glycol, tetramethylene glycol, 2,3-butylene glycol, triethyl phosphate, polyvinyl alcohol, partially hydrolysed polvinyl acetate, polyacrylic acid and polyvinyl pyrrolidone.

19. A composition according to any one of the preceding claims, wherein the plasticizer comprises a water-soluble and a water-insoluble plasticizer.

20. A composition according to any one of claims 1 to 10, 13, 14, 17 and 18, wherein both the inert filler and the plasticizer are soluble in water.

21. A composition according to claim 1 substantially as hereinbefore described.

22. Process for the production of shaped articles, which comprise shaping a composition claimed in any one of claims 1 to 21.

23. Process according to claim 22, wherein the composition is shaped by injection moulding, vacuum forming, extrusion or calendering.

24. Process according to claim 22 or 23, wherein the composition is subjected to cold pressing.

25. Process according to any one of claims 22 to 24, wherein part or all of the plasticizer is removed from the shaped articles by solvent extraction.

26. Process according to claim 25, wherein part or all of the plasticizer or filler or both is extracted with water.

27. Process for the production of shaped articles according to claim 22 substantially as hereinbefore described.

28. Shaped articles when produced by the process claimed in any one of claims 22 to 27.

29. Process for the production of a composition claimed in any one of claims 1 to 21, which comprises milling a dry blend of the components thereof.

30. Process for the production of a composition claimed in any one of claims 1 to 21, which comprises adding the filler to the fluxed polymer, adding plasticizer if required, and milling the resultant mixture.

31. Process according to claim 30, wherein the filler is initially in the form of a concentrate in part of the polymer and the concentrate is diluted with the remainder of the polymer and the plasticizer.

32. Process for the production of a composition claimed in any one of claims 1 to 21 substantially as hereinbefore described.

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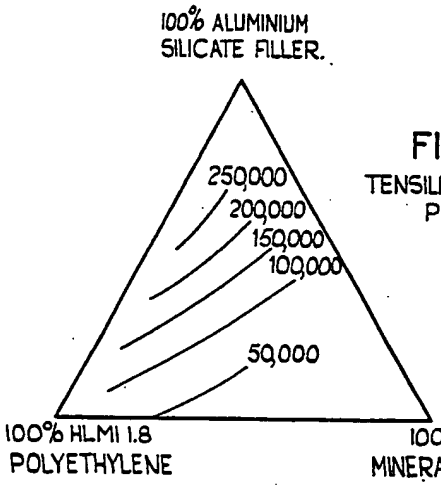

FIG.1.
TENSILE MODUI
PSI.

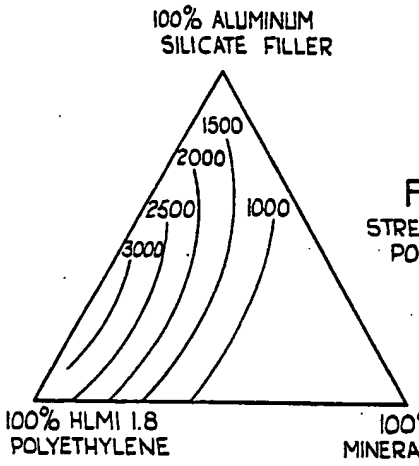

FIG.2.
STRESS AT YI
POINT, PSI

1044028 COMPLETE SPECIFICATION
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Sheets 1 & 2

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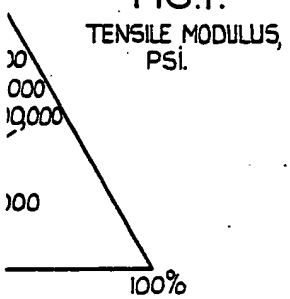
FIG.1.
TENSILE MODULUS, PSI.



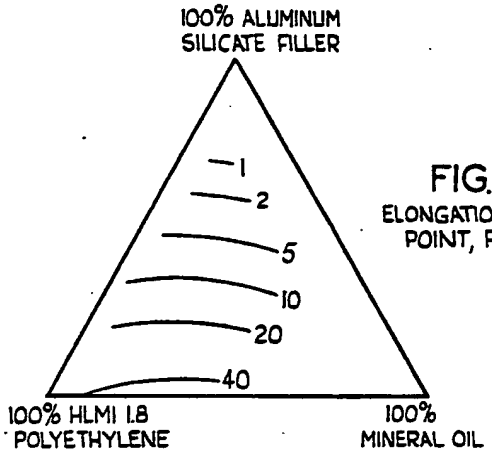
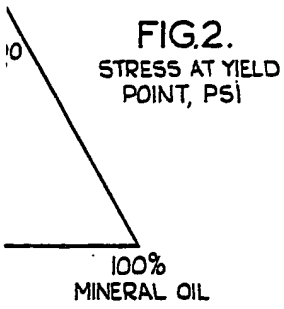


FIG.3.
ELONGATION AT YIELD POINT, PERCENT.

NUM
ILLER

FIG.2.
STRESS AT YIELD POINT, PSI



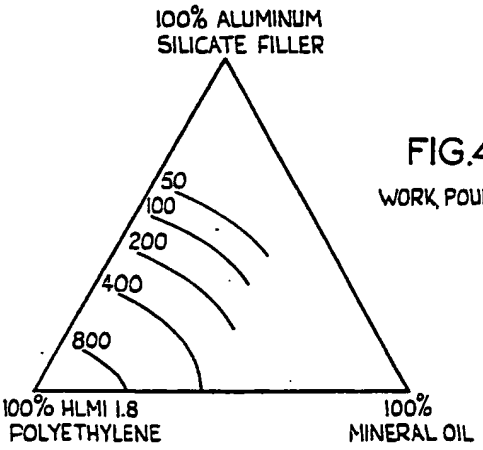


FIG.4.
WORK, POUND/INCH

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Sheets 1 & 2

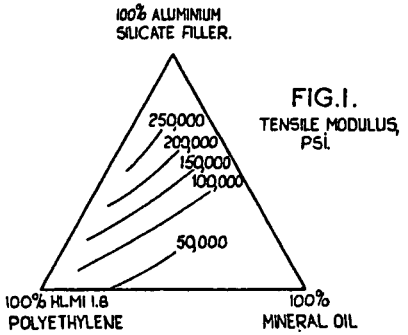

FIG.1.
TENSILE MODULUS, PSI.

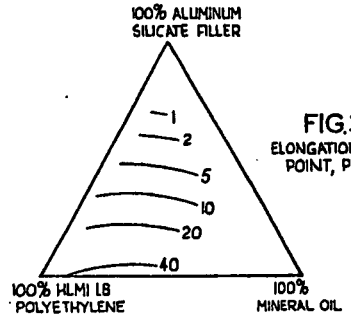

FIG.3.
ELONGATION AT YIELD POINT, PERCENT.

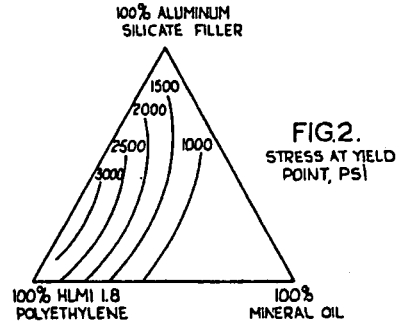

FIG.2.
STRESS AT YIELD POINT, PSI

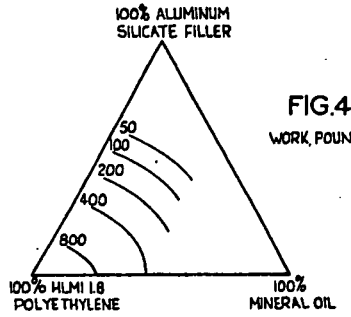

FIG.4.
WORK, POUND/INCH

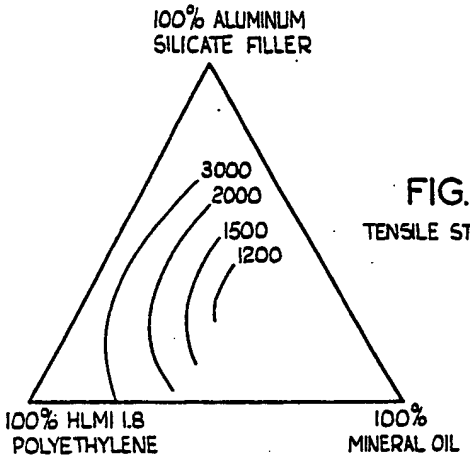

FIG.5.
TENSILE STRE

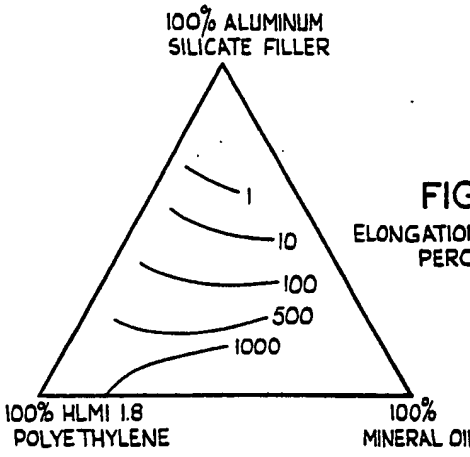

FIG.6
ELONGATION
PERCEN

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 3 & 4

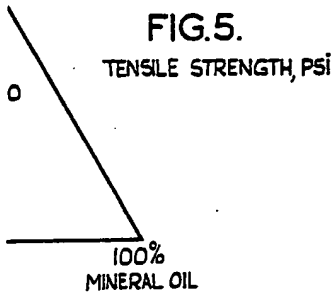

FIG.5.
TENSILE STRENGTH, PSI

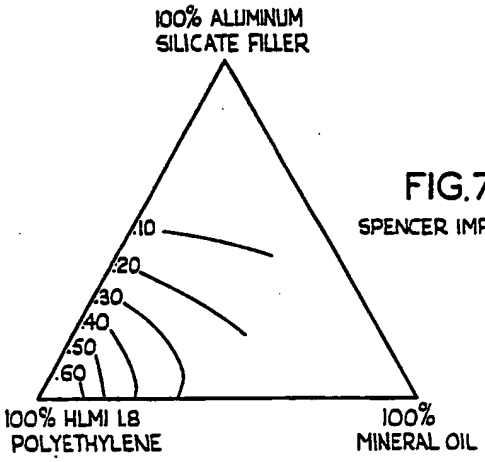

FIG.7.
SPENCER IMPACT, PSI

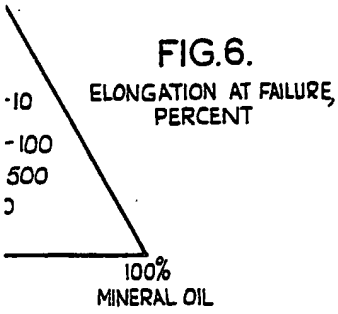

FIG.6.
ELONGATION AT FAILURE, PERCENT

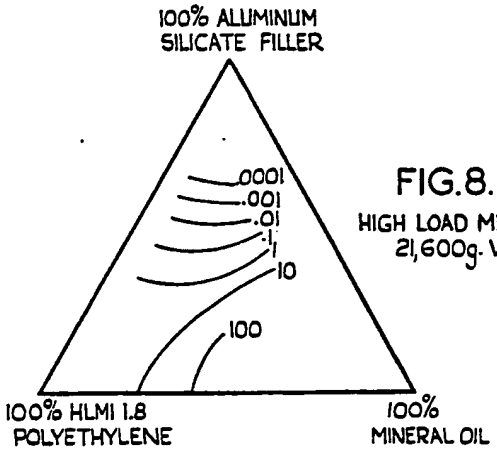

FIG.8.
HIGH LOAD MELT INDEX
21,600g. WT.

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 3 & 4

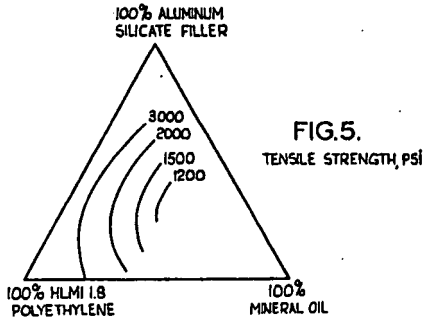

FIG.5.
TENSILE STRENGTH, PSI

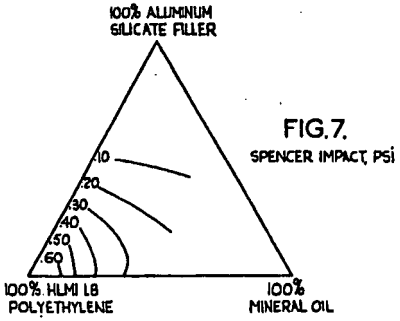

FIG.7.
SPENCER IMPACT, PSI

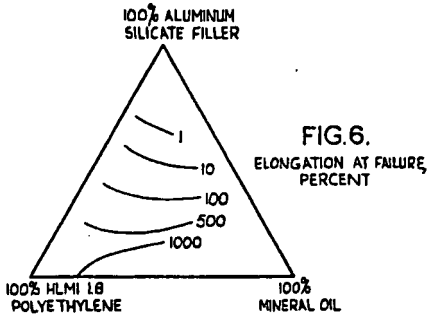

FIG.6.
ELONGATION AT FAILURE, PERCENT

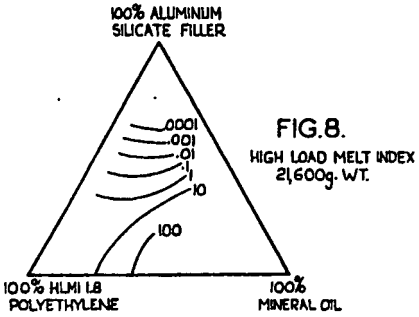

FIG.8.
HIGH LOAD MELT INDEX 21,600g. WT.

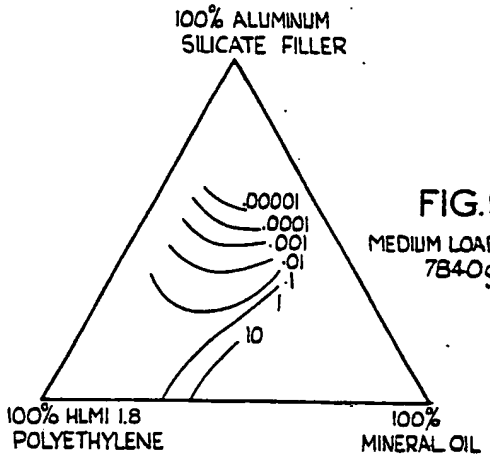

FIG.9.
MEDIUM LOAD MELT
7840g WT.

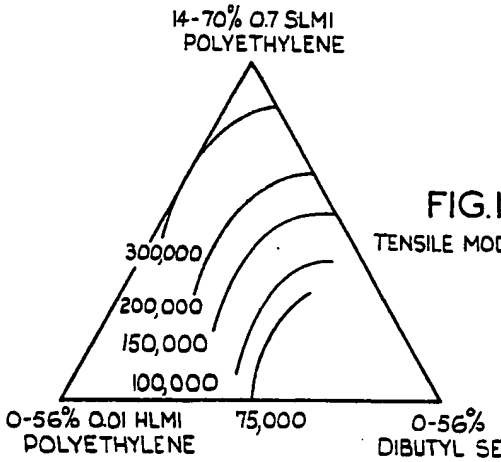

FIG.10.
TENSILE MODULUS

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 5 & 6

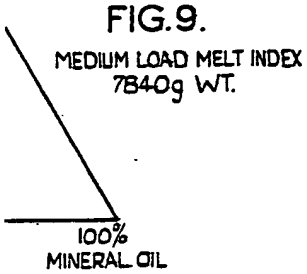

FIG.9.
MEDIUM LOAD MELT INDEX
7840g WT.

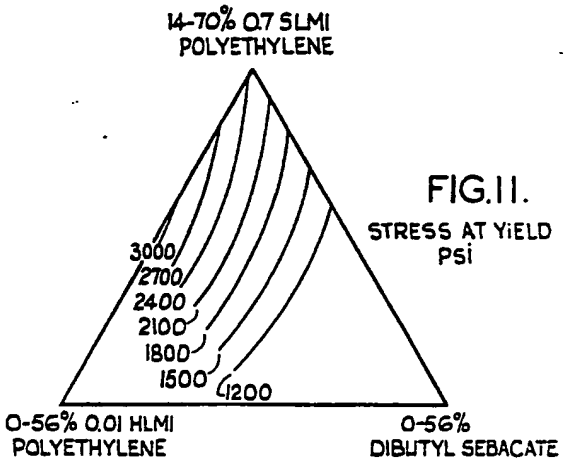

FIG.11.
STRESS AT YIELD POINT
PSI

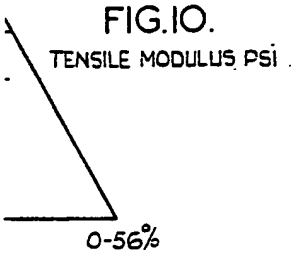

FIG.10.
TENSILE MODULUS PSI

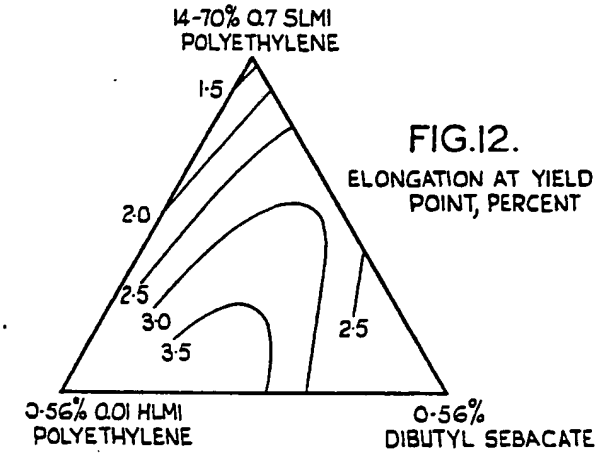

FIG.12.
ELONGATION AT YIELD
POINT, PERCENT

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 5 & 6

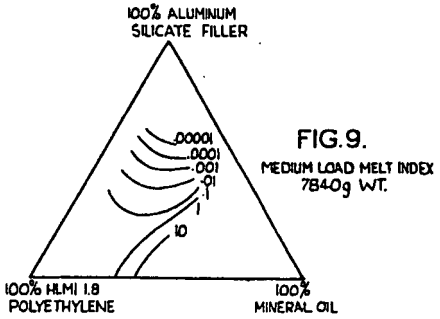

FIG.9.
MEDIUM LOAD MELT INDEX 7840g WT.

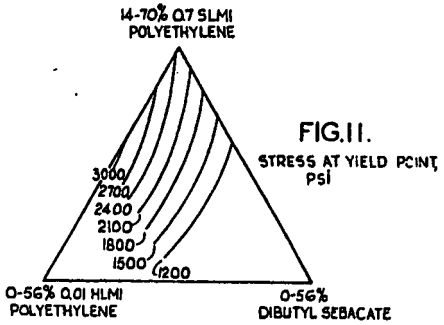

FIG.11.
STRESS AT YIELD POINT, PSI

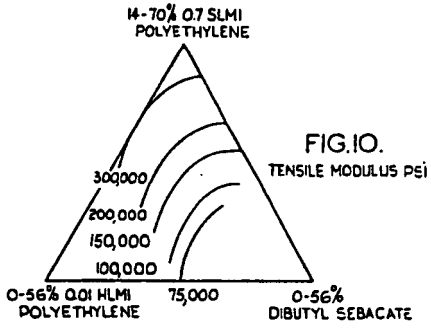

FIG.10.
TENSILE MODULUS PSI

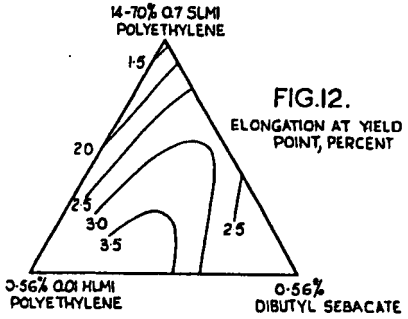

FIG.12.
ELONGATION AT YIELD POINT, PERCENT

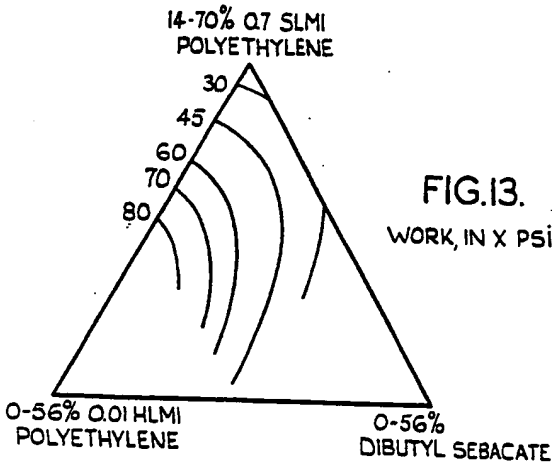

FIG.13.
WORK, IN X PSI

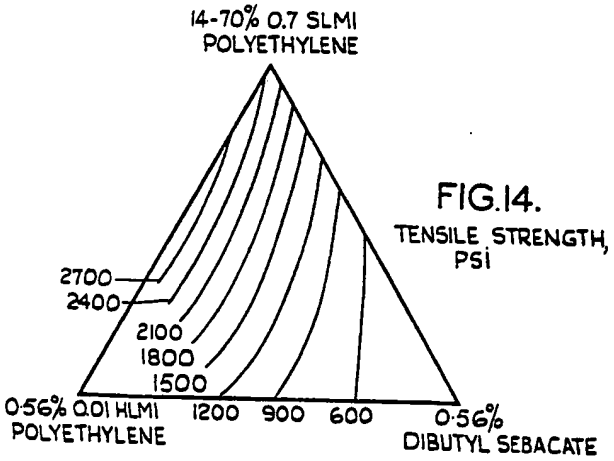

FIG.14.
TENSILE STRENGTH, PSI

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 7 & 8

G.13.
IN X PSI

SEBACATE

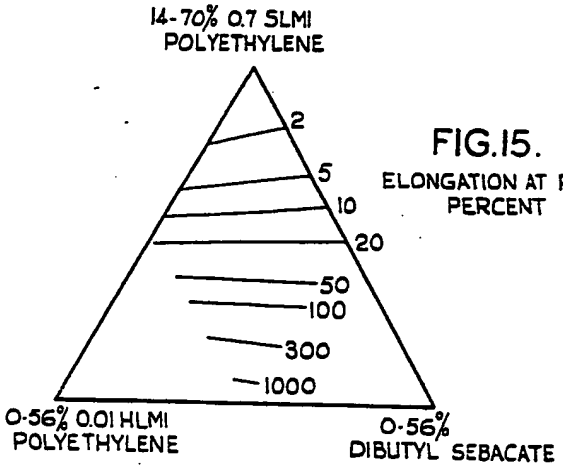

FIG.15.
ELONGATION AT FAILURE, PERCENT

G.14.
E STRENGTH, SI

16%
YL SEBACATE

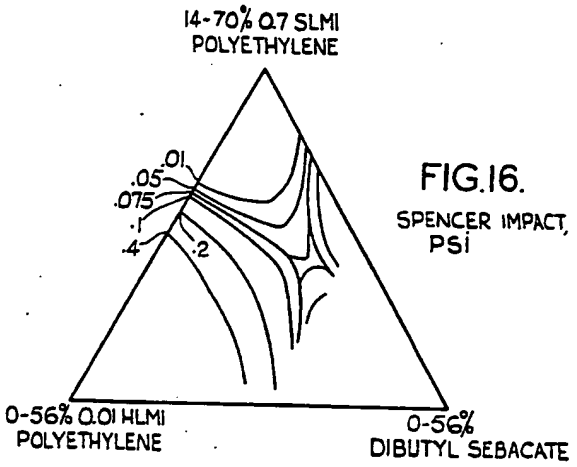

FIG.16.
SPENCER IMPACT, PSI

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 7 & 8

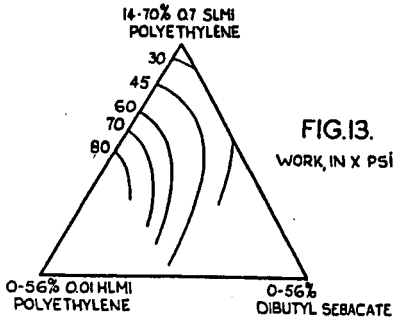

FIG.13.
WORK, IN X PSI

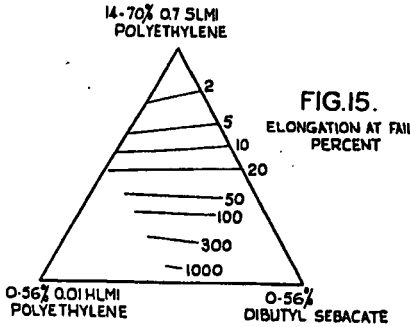

FIG.15.
ELONGATION AT FAILURE, PERCENT

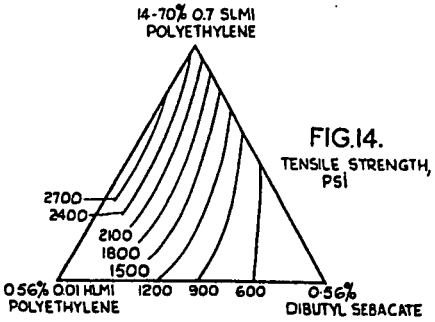

FIG.14.
TENSILE STRENGTH, PSI

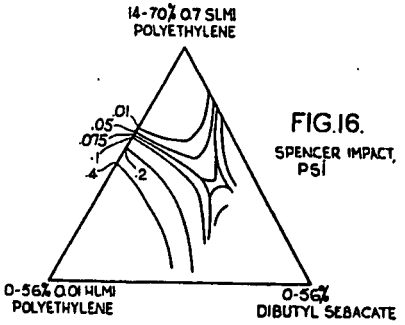

FIG.16.
SPENCER IMPACT, PSI

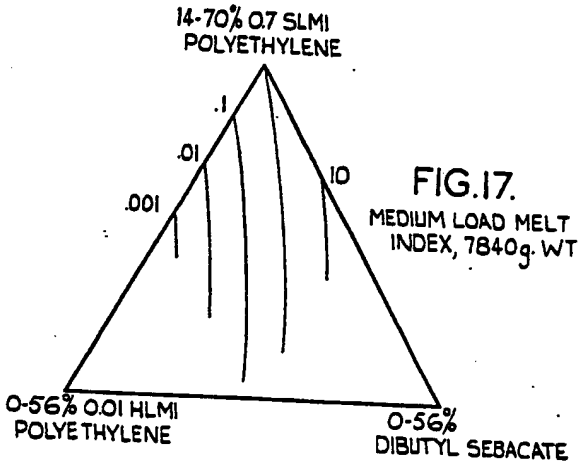

FIG.17.
MEDIUM LOAD MELT INDEX, 7840g. WT

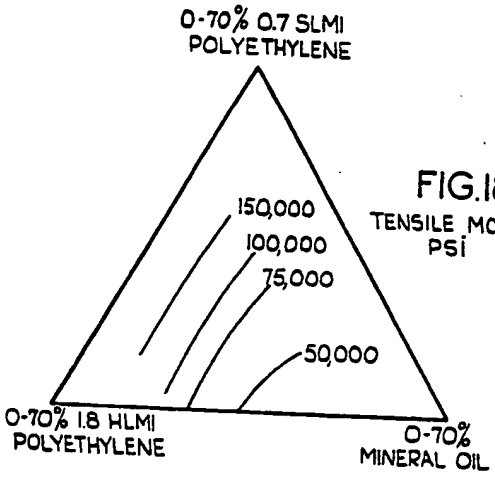

FIG.18.
TENSILE MODULUS, PSI

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 9 & 10

IG.17.
M LOAD MELT
EX, 7840g. WT

6%
YL SEBACATE

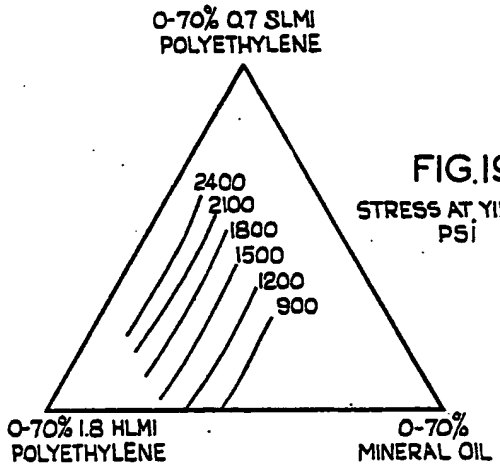

FIG.19.
STRESS AT YIELD POINT, PSI

G.18.
MODULUS, SI

0%
OIL

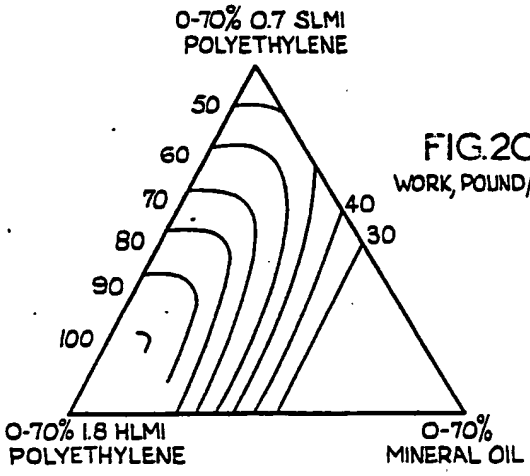

FIG.20.
WORK, POUND/INCH

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 9 & 10

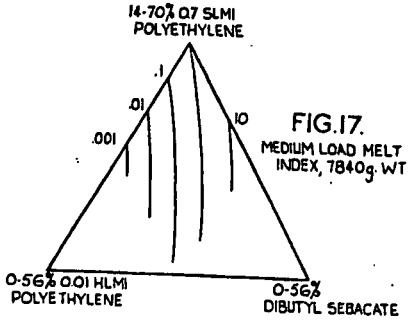

FIG.17.
MEDIUM LOAD MELT INDEX, 7840g. WT

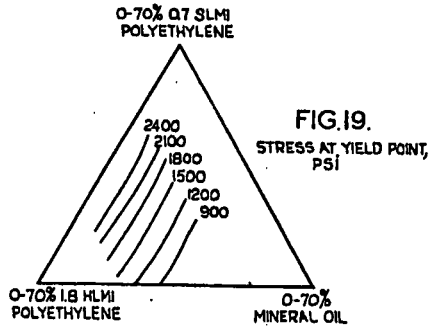

FIG.19.
STRESS AT YIELD POINT, PSI

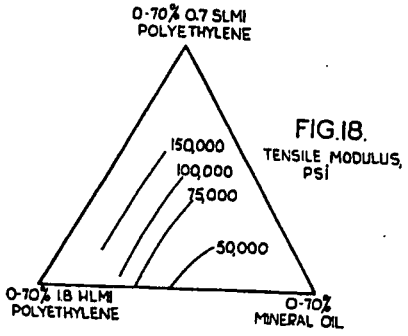

FIG.18.
TENSILE MODULUS, PSI

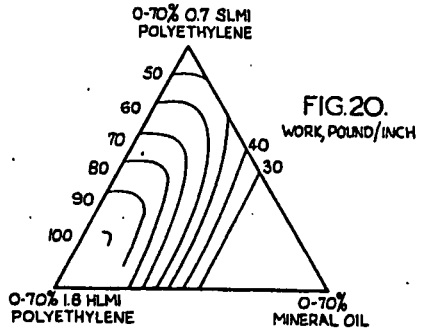

FIG.20.
WORK, POUND/INCH

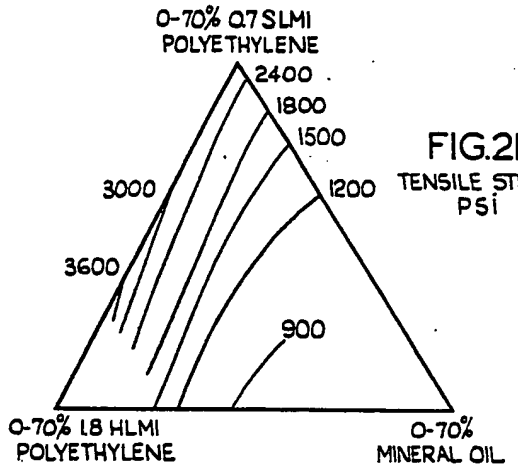

FIG. 21.
TENSILE STRENGTH, PSI

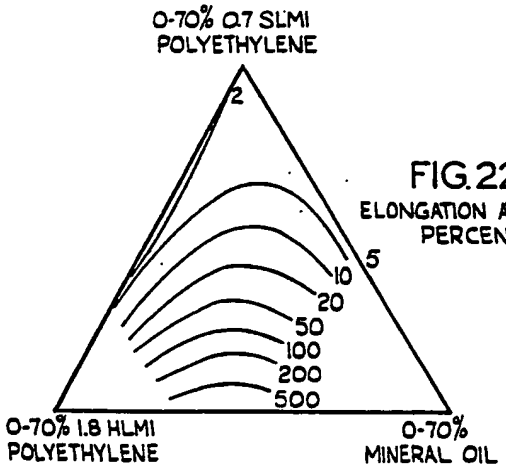

FIG. 22.
ELONGATION AT FAILURE, PERCENT

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 11 & 12

IG.21.
ILE STRENGTH,
PSI

0%
L OIL

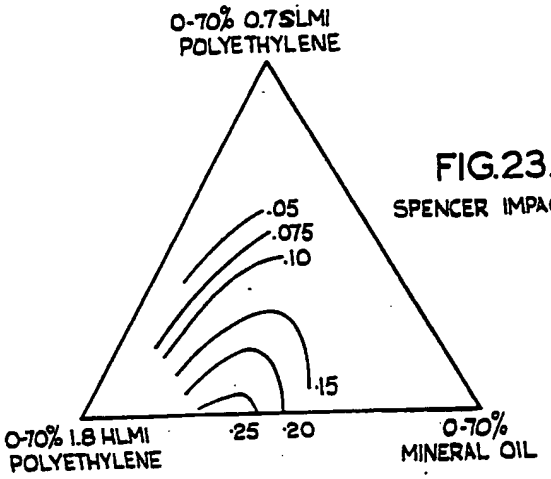

FIG.23.
SPENCER IMPACT, PSI

3.22.
ION AT FAILURE,
RCENT

%
OIL

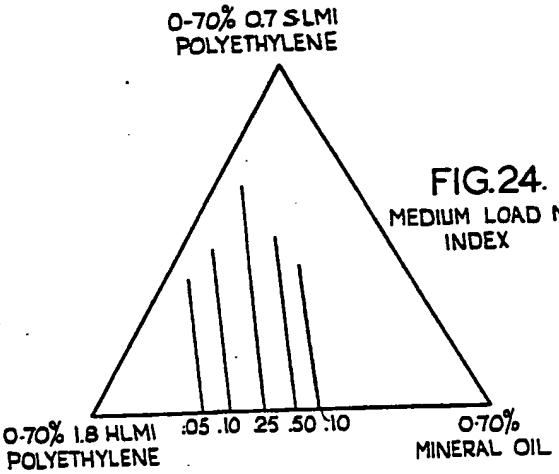

FIG.24.
MEDIUM LOAD MELT
INDEX

1044028 COMPLETE SPECIFICATION
12 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 11 & 12

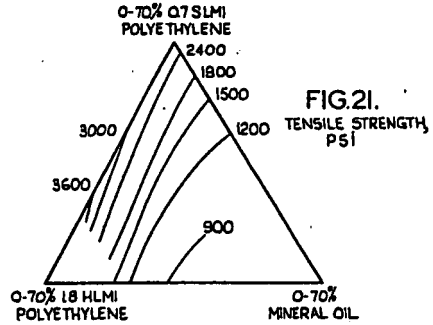

FIG.21.
TENSILE STRENGTH, PSI

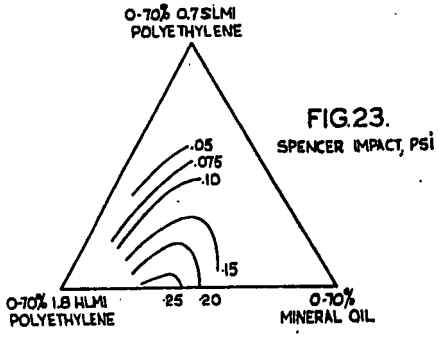

FIG.23.
SPENCER IMPACT, PSI

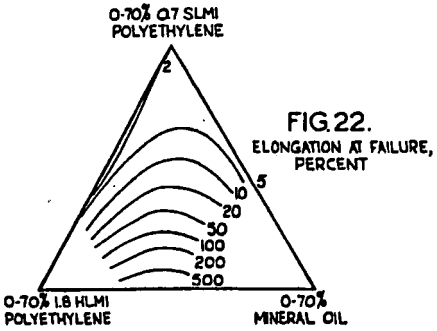

FIG.22.
ELONGATION AT FAILURE, PERCENT

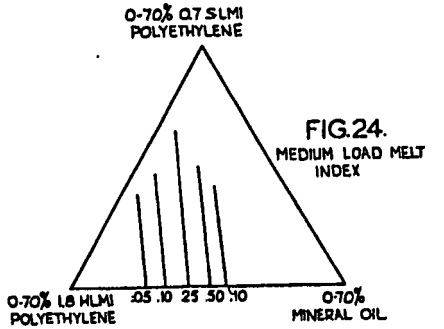

FIG.24.
MEDIUM LOAD MELT INDEX